TEST METHODS

OVERVIEW
During product development of metalworking fluids, many tests are run to determine the physical, chemical and performance properties of the fluid which are often compared to a previous product or a competitive fluid.

The intent of the test method is to provide predictive results for performance testing and physical/chemical results for later analysis so that a newly developed fluid meets the criteria of the customer or market and can be analyzed during use.

Different product lines (machining fluids, cleaners, corrosion preventives, etc.) employ different methods depending upon what is required and/or accepted for their industries. There are also some larger global customers that have their own test methods. These methods provide some amount of internal consistency within a customer database. Sometimes tests must be certified by a third-party laboratory in order to minimize any bias. Many automotive customers and tier two suppliers maintain these types of rules.

DEFINITION
American Society for Testing and Materials (ASTM), Society of Manufacturing Engineers (SME) and Society of Automotive Engineering (SAE) are three United States organizations that provide different test methods. Also, there are many standards that make their way into the United States because of globalization.

There are many DIN standards that are used. DIN is German for Deutsches Institut fur Normung. In English this reads as German Institute for Standardization. While DIN is German in origin, since 1975, Germany represents interests at the international and European levels. There are also International Standards Organization (ISO) standards that are used. Often a method from one organization will reference the competing method from another organization.

A test method should contain the necessary details to make sure it is a valid method. Details to consider for an effective test method are:

» References or appropriate sources
» List of hazards or safety conditions
» Interferences with the method
» List of reagents and apparatus required to run the method
» Detailed step-by-step procedure
» Detailed calculations
» Repeatability and reproducibility data (R&R) to show the variance
» Limits of detection or any particular sensitivities

Particular care should be taken to interpret results as they might apply to the particular industry.
LIST OF METHODS

While there are many different methods available, listed below are some of the more common types. Again, some are particular to specific product types or industries.

Acid Number or Acidity – measures the total acidity of a product to confirm the correct amount of fatty acids or the lack of acid-bearing compounds in a straight oil. Free acids can also be assessed in metalworking fluid.

Acid Split – measures the total amount of active materials that can be split from an emulsion. For most fluids, these are the non-water soluble materials like oil, ester and fatty acids. This can be used as a concentration control method but the influx of tramp oil will impede accurate readings.

Alkalinity – measures the total amount of alkaline materials. Different methods permit for total alkalinity versus reserve alkalinity. Alkalinity is important relative to maintaining biostability and corrosion protection. Alkalinity and pH, while related, are not the same. Low alkalinity does not always mean low pH and visa versa.

Appearance – how the product appears under different temperatures or conditions.

Bioresistance – standard 8-week ASTM test to assess a fluid's resistance to bacteria and fungi, separately. Additional testing can be conducted to examine the two organisms together.

Cloud Point – measures the temperature at which a certain chemical will no longer be soluble and it will come out of solution and create a cloudy appearance.

Cobalt Leaching – for customers that utilize a lot of carbide tooling, cobalt is the binder that holds the carbide together. This method assesses the degradation of the carbide by monitoring cobalt content.

Conductivity – ability of a liquid to transfer electricity; for most water-soluble products, ions in the water impact the conductivity. For some emulsions, high levels of conductivity can actually disrupt the emulsion and create a split condition.

COD/BOD – Chemical Oxygen Demand is the measurement of all chemicals in the water which can be oxidized. The Biological Oxygen Demand measures oxygen uptake by bacteria during the oxidation of organic matter. Both tests are often run on the water phase after waste treatment to determine the efficiency of the waste treatment process.

Cold Test – temperature at which a particular fluid may solidify or create a gel; result is used to assess a pass or fail.

Color – like appearance, this is strictly a visual examination to make sure the product appears as it should. There are color standards for comparison.
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**Corrosion** – there are several methods used for ferrous corrosion testing; there are a few types of cast iron chip tests as well as tests that use steel panels; there is also a stack stain test generally used for corrosion preventives.

**Density** – required for the SDS; it is the mass/volume calculation; it is also reported as lbs/gal.

**Dirt Content** – test used to assess used fluids with regard to how much “soil” they contain. The soil can be fines and other debris that can be filtered at a specific micron level to confirm how much “dirt” is present. This is used for comparative testing.

**Elastomer Compatibility** – this ASTM procedure examines elastomers after their exposure to a fluid under controlled conditions; the changes examined are hardness and size.

**Elemental Analysis** – many times specific elements have to be acknowledged as being present or absent in a product. Typical elements assessed are boron, calcium, chlorine, magnesium, phosphorus, sodium, silicon and sulfur.

**Emulsion Stability** – just about every water-soluble fluid will get exposed to the customer’s water to confirm that the dilution (solution or emulsion) is stable; sometimes the testing is performed under dynamic conditions with specific contaminants.

**Flash Point & Fire Point** – flash point is required for the SDS and some customers want to see the fire point as well. There are different methods depending upon the boiling point of the fluid which is driven by the water content.

**Foam** – there are several methods to examine foam, but the best methods generally use some type of recirculation. Many customers use a blender foam test but blenders run at 17,000 rpms which is not representative of most fluid pumps. The foam test should examine the foam build and foam breaking.

**Lubrication** – there are many test methods for this parameter, including Falex Pin and V-Block, Falex Four Ball, Twist Compression Test, Tapping Torque Test, Coefficient of Friction Test and others; the test that most closely replicates the lubrication regime of the customer process should be used. Lubrication testing using an actual CNC machine with the same metal and tool used in the process can give an excellent approximation of the effectiveness of the product. A standard product or the product currently in use should be analyzed at the same time as the test product.

**Microbiological** – for fluids that must maintain long sump lives, resistance to bacterial and fungal growth is important. Two methods are used to determine the presence of growth: plate counts (auger in a petri dish) and dip slides. Both methods report results a Colony Forming Units per milliliter (CFU/ml). Dip slides have become popular because of the “ease of use” in the field. Proper storage of unused slides is important for accurate assessments.
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**Moisture** – some fluids are sensitive as to the presence of water. Two methods are distillation and the Karl Fisher (KF) titrator. The distillation is used for larger quantities of water while the KF titrator is used for trace amounts.

**Neat Product Stability** – similar to emulsion stability in that the neat product is assessed for stability at four basic temperatures: 0, 40, 75 and 120°F. The testing should include the most severe conditions under which the fluid may be stored. Some products are unstable when frozen or heated too high.

**Non-ferrous Staining** – similar to corrosion testing but is reserved for non-ferrous alloys such as cast aluminum, wrought aluminum, copper, brass and bronze. Appropriate metal samples are submerged in fluid and the metal surface below the fluid level, at the fluid level and above the fluid level (vapor phase) are examined for stain.

**Odor/Smell** – another requirement for the SDS. Helps identify the fluid but can be somewhat subjective.

**pH (Neat and Diluted)** – another requirement for the SDS. The diluted pH is also important relative to staining, corrosion and is particularly critical to maintaining bioresistance.

**Pounds per Gallon** - (see Density)

**Pour Point** – temperature at which a fluid will no longer pour. Useful for products that might have a tendency to freeze but not split.

**Refractometer** – while many end users prefer to use the hand held refractometer for concentration control, this method is not very accurate and can be misleading.

**Residue Characteristics** – this is similar to emulsion characteristics in that the dried fluid should not be tacky or sticky and should be resoluble in its own dilution or water.

**Saponification** – this method is referred to as “SAP” and is used for fluids that contain fats or esters. Fat and esters can be saponified and then titrated to determine the amount.

**Stack Stain** – this method attempts to duplicate what happens when steel surfaces are sandwiched together with a liquid between them. Often times this method is run at elevated temperatures to hasten the results.

**Surface Tension** – caused by the cohesive forces between liquid molecules that tend to restrain the liquid from flowing or wetting a surface. The surface tension value is useful to predict a cleaner’s ability to wet out soils on a surface and may also impact lubrication or foam.

**Tramp Oil Rejection** – this method is used to assess the amount of oil a fluid will reject after being contaminated with that oil. The level of rejection is also dependent upon the level of shear used to mix the oil into the fluid. Tramp oil determination can become complex when multiple sources of tramp oil are present.
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Viscosity – The resistance to flow by a liquid. Generally this is used on straight oils or product concentrates.

Volatile Organic Compound (VOC) – determines how much of the fluid will “evaporate” into the air based on the temperature of the test. The EPA method uses 110°C for the evaporation test, but some customers prefer temperatures closer to the operating conditions of the application.

CONCLUSION

It is important to know what test methods may be involved with your customer’s decision making process, what the test procedure details are and what the results of the method tell the customer. Understanding various metalworking test methods will help in the product recommendation process and in management of the systems once the business is obtained.